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(54) INORGANIC MEMBRANE FOR MICROFILTRATION, AND A PROCESS FOR PRODUCTION OF SUCH AN INORGANIC MEMBRANE

ANORGANISCHE MEMBRAN FÜR MIKROFILTRATION UND VERFAHREN ZU IHRER HERSTELLUNG

MEMBRANE MINERALE DE MICROFILTRATION ET SON PROCEDE DE PRODUCTION

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- **NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH vol. B19/, no. III?, February 1987, pages 204 - 208 P. SIOSHANSI 'MEDICAL APPLICATIONS OF ION BEAM PROCESSES'**

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Description

The present invention relates to an inorganic membrane for microfiltration, that consists of a macroporous inorganic support and an inorganic membrane layer with pores having a pore size typically between 0.01 μm and 10 μm, which membrane layer is connected rigidly to the inorganic support. The invention relates also to a process for production of such an inorganic membrane.

A membrane consisting of a macroporous support and a membrane layer is a composite membrane. The macroporous support contributes in here to the mechanical strength of the total membrane. Whenever the membrane layer with a high pore density is deposited relatively thin in comparison to the mean pore size on a relatively thick support a membrane with a high flux is obtained. In case the inorganic membrane layer as well as the inorganic support is made of suitable ceramic materials then the membrane will be chemically highly inert and will operate well at elevated temperatures.

From EP-A-0-144 097 a composite ceramic membrane is known having above mentioned characteristics. Said ceramic membrane has a membrane layer that is formed by coating a porous support with a thin film by immersion in a stable sol of colloidal metal oxide (g - ALOOH) particles and then drying and heating the support, thus forming a microporous membrane layer of metal oxide particles.
Such known inorganic membranes may display mechanical faults, like little fractures and 'pinholes' with typical size 10 μm to 100 μm, which are relatively large in comparison to the mean pore size, typically 5 nm - 5 μm, thus reducing the separating characteristics.

Another intrinsic disadvantage of these membranes is the relatively large thickness of the membrane layer in comparison to the mean pore size, resulting in reduced flux characteristics of the membrane. In said patent publication the thickness of the membrane layer ranges from 50 to 1000 times the mean pore size of the membrane layer.

The present invention has as an object to provide an inorganic membrane with improved separating and flux characteristics. An other object is to provide an inorganic membrane particularly useful for biomedical filtration, in particular in solutions with vulnerable cells. Therefore, according to the invention an inorganic membrane for microfiltration is proposed, wherein the membrane consists of an inorganic macroporous support having a mean pore size in the range of 0.25μm-25μm, and an inorganic membrane layer being deposited and connected rigidly to a flattened surface of the macroporous support by means of a deposition method, whereby the pores of the membrane layer are perforations shaped as shallow channels square to the membrane layer, and are formed with a diameter in the range of 0.05-5μm by means of etching, said channels having a length at least smaller than ten times the diameter of the channels.

Good filtration results are obtained according to the invention with an inorganic membrane consisting of a porous support of alumina with mean pore size in the range 0.25-25 μm and an inorganic membrane layer of silicondioxide with channels of 0.05-5 μm In preference the support and the membrane layer are constituted from equivalent materials with the same inorganic components. An inorganic membrane is thus obtained appplicable in a broad temperature range with a good cohesion between the support and the membrane layer. Because of the low length/diameter ratio of the channels an inorganic membrane with a high flux is obtained. In addition through this measure vulnerable cells have a minimum chance in being damaged during passing these channels.

The inorganic membrane may be produced by firstly depositing a suitable inorganic layer by chemical vapour deposition on the inorganic support, and secondly making perforations in this inorganic layer by means of a suitable etching process.

According to the invention an inorganic membrane is manufactured, in which the perforations in the membrane layer are made by means of a lithographic etching process, in particular UV lithography in combination with chemical etching. On the inorganic layer a photo-lacquer layer is formed, this photo-lacquer layer being exposed to a regular mask pattern with the use of a suitable source, this photolacquer layer being developed, then in the inorganic layer the mask pattern is etched by a suitable wet or dry etchant, thus forming the membrane layer. The use of a lithographic etching process offers the advantage that the shape of the cross-section of the channels may be designed according to demand. Long shaped rectangular crosssection will lead to high flux.

The pattern of the membrane layer may be given any particular design, practically with a repetition rate in the range 0.1-10 μm. It is thus conceivable to separate not only particles according to their size, but also to their shape. This may be particularly usefull for the separation of biological cells, e.g. yeast or blood cells.

Appropriate materials for the membrane layer of the membrane are according to the invention composed of ceramic constituents like silicondioxide, siliconnitride, siliconcarbide, alumina, zirconiumoxide, titaniumoxide and titaniumnitride or other metaloxides, nitrides or silicides. Other materials like carbon, silicon, or metals like gold, silver, chromium, nickel, aluminum, titanium etc. are also appropriate. For biomedical microfiltration, e.g. separation of blood components, biocompatibel or bloodcompatibel materials may be used like titanium, titaniumnitride, chromiumalloys, carbon, siliconcarbide, silicon, siliconnitride or other appropriate semiconducting materials and noble metals.

According to the invention a process for production of the inorganic membrane is proposed, the membrane consisting of a macroporous inorganic support and an inorganic membrane layer whereby an inorganic layer is deposited on a flattened surface of the support by means of chemical vapour deposition or sputtering, and whereby on the inorganic layer a photo-lacquer layer is formed, this photo-lacquer layer being exposed to a reg-

ular mask pattern with the use of a suitable source, this lacquer layer being developed, thereafter in the inorganic layer the mask pattern is etched by an etchant, forming perforations shaped as channels, square to the inorganic layer, thus forming the inorganic membrane layer. By depositing the flattening material on the porous support a reasonable uniform flat and dense surface is obtained. Useful deposition methods are vapour deposition, chemical vapour deposition, sputtering, sol/gel coating for small pores and spray coating, sol/gel coating, use of rubbing compouds, etc. for large pores. The inorganic layer deposited on a flat and dense surface will exhibit an almost featureless surface morphology, whilst the microstructure of an inorganic layer deposited directly, e.g. by means of chemical vapour deposition, on the porous support may consist of clusters of grains tightly packed together. A flat surface morphology of the inorganic layer is important in relation to the precision in which the channels may be formed by the subsequent lithographic etching. Materials like silicon and aluminum are suitable as a flattening material, especially polysilicon is well capable in filling pores. The flattening material should in a later stage of the process be removed from the pores in the support in order to obtain a permeable membrane. In preference the flattening material is removed by means of a material selective etchant that is brought into contact with the flattening material through the yet formed membrane layer. The selective etchant should mainly attack the flattening material and hardly the support material or the membrane layer.

Another preferential process in producing the membrane according to the invention is characterized in that subsequent to the deposit of the flattening material on the support a supplementing flattening process is being performed. The flattening process may be a mechanically polish method. The flattening process may also according to the invention be characterized in that on a surface of the flattening material firstly a thin lacquer layer is deposited, which lacquer layer will be hardened such that a very smooth surface is obtained. Secondly the hardened lacquer layer with a smooth surface is being etched, such that all lacquer layer will be removed, including part of the flattening material and the support, thus obtaining a very smooth and dense support surface with filled pores. The etching of the lacquer layer may be achieved by reactive ion (plasma)etching.

EP-A-0.325.752 discloses an inorganic membrane for microfiltration, wherein the membrane consists of a macroporous inorganic support and an inorganic membrane layer with pores having a pore size, typically between 0.01μm and 10 μm, which membrane is connected rigidly to the inorganic support, whereby the pores of the membrane layer are perforations shaped as shallow channels, square to the membrane layer, in which the channels have a length and a diameter, such that the length is at least smaller than ten times the diameter of the channels. An intrinsic disadvantage of this membrane is the relative mechanical weakness of the membrane layer due to the deep orthogonal grid of grooves, being at least one half thickness of the membrane layer. Also the operation of the laser apparatus is very critical with respect to the thickness of the membrane layer, the material of the membrane layer, power intensity. Without a suitable mask pattern on the membrane layer the process of etching is very critical with respect to vibrations.

FR-A-2.596.289 describes a process for the production of a gas filtration membrane layer using a flattening material.

The invention will now be explained by means of the accompanying drawing in which:

Figure 1 shows in cross-section a part of the inorganic membrane according to the invention.
Figures 2 and 3 show in cross-section part of the inorganic membrane according to some preferential examples according to the invention.
Figures 4 to 9 show in cross-section subsequent stages of a process for production of the inorganic membrane according to the invention.
Figures 10 to 16 show in cross-section subsequent stages of a preferential process for production of the inorganic membrane according to the invention.

The figures are schematized and are not drawn on scale. Similar parts may have the same reference-mark.

Figure 1 shows schematic in cross-section a part of the inorganic membrane according to the invention. The inorganic membrane consists of a macro-porous inorganic support 1, in this example a wafer of α-alumina with thickness 2.5 mm and mean pore size 5 μm and an inorganic membrane layer 2 of silicondioxide with thickness 2 μm. The perforations in the membrane layer 2 are shaped as shallow channels 3, square to the membrane layer 2, in which the channels 3 have a length at least smaller than ten times the diameter of the channels 3. The channels 3 are formed here with a diameter of 1 μm in the membrane layer 2. The length of the channels 3 is here two times the diameter of the channels 3. The shape of the cross-section of the channels 3 is here circular. Depending on the application other shapes for the cross-section of the channels 3 may be chosen. For example rectangular shape, figure 2 or with line pattern shape, figure 3. Rectangular shape has the advantage that particles have difficulty in closing the channels totally. Line pattern shape of the membrane layer has the potential of a high flux. Circular or round shape of the cross-section has an advantage in separating media with vulnerable particles, in particular shallow channels with a rounded and smooth morphology are appropriate for separating biological cells.

Figures 4 to 9 show in cross-section subsequent stages of a process for production of the inorganic membrane consisting of a macroporous inorganic support and an inorganic membrane layer according to the invention. Prior to the deposition of the inorganic layer 4 the pores lying at the surface of the inorganic support have been flattened and filled by rubbing for example a fine

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siliconpowder 7 on the surface, and if necessary followed by a light polishing method, figure 4. On the flattened surface of the inorganic support 1, figure 4, in this example a wafer of siliconcarbide with thickness 2.5 mm and mean pore size 15 µm, an inorganic layer 4 of siliconnitride with thickness 3 µm is deposited by means of a suitable deposition method, here by means of 'Chemical Vapour Deposition', figure 5. The inorganic layer 4 is formed by chemical deposition of dichlorosilane and ammonia at low pressure (LPCVD). In the inorganic layer 4 then perforations are formed through the use of a lithographic determined etching process. On the inorganic layer 4 a photo-lacquer layer 5 is formed, figure 6, in this example Eastman Kodak Resist KPR-820. The photo-lacquer layer 5 is then being exposed to a regular pattern, figure 7, with the use of a suitable UV source, here with a Nikon NSR-1010i3 projection system and with the use of a mask 6 that is projected five times reduced. The pattern of the mask is made here of round fields with a diameter of 15 µm. Subsequent the lacquer layer 5 is developed and etched leaving a reduced(5x) mask pattern of photo-laquer 5 on the inorganic layer 4, figure 8, according to standard lithographic methods. In the inorganic layer 4 then the reduced mask pattern is etched by a suitable etchant, forming perforations with a diameter of 3 µm, herewith forming the inorganic membrane layer 4, figure 8, 9. In this example the inorganic layer 4 of siliconnitride is etched by means of a stabilized fluorohydrogen (HF) solution. Perforations, shaped as shallow channels 3 with circular cross-section are thus formed in the inorganic layer 4. Through these yet formed channels 3 the silicon powder 7 is selectively etched with tetramethylammoniumhydride. Finally all lacquer residues are removed and a clean inorganic membrane layer 4, figure 9, is obtained.

Figures 10 to 16 show in cross-section subsequent stages of a preferential process for production of the inorganic membrane according to the invention. Before the inorganic layer 4 is deposited on a surface of the support 1, figure 10a, primarily a suitable flattening material 7 is deposited on the surface of the support 1 filling all pores lying at the surface of the support, 1, figure 10b. The thickness of the flattening material 7 is related to the mean pore size of the porous support 1, in order to fill all pores lying at the surface of the support 1. In this example a porous support 1 of alumina with mean pore size 2 µm has as flattening material 7 a polysilicon layer with a thickness of about 5 µm. The polysilicon layer 7 is deposited on the support by disintegretion of silane (SiH4) at low pressure (LPCVD). Other materials like alumina with a well porefilling capability may also be used. After the polysilicon deposition in preference a supplementing flattening process is performed. A mechanical polish with diamond powder will do very well.

In preference according to the invention subsequent to the deposition of the flattening material 7 on the support 1 the flattening process is firstly performed by depositing on a surface of the flattening material 7 a thin lacquer layer 8, figure 11. For a thin, uniform and smooth lacquer surface 9 the method of 'spin-coating' may be employed. Next the lacquer layer 8 is hardened yielding a very smooth surface 9. Then the hardened lacquer layer 8 is etched, such that all lacquer layer 8 will be removed, including part of the flattening material 7 and the support 1, herewith obtaining a very smooth and dense support-surface 10 with filled pores, figure 12. The lacquer layer 8, the flattening material 7 and a part of the support 1 is well etched with a plasma containing tetrafluoromethane(CF_4) and oxygen, resulting in a very smooth surface 10 with an overall surface roughness less than 0.1 µm.

Next an inorganic layer 4 of silicondioxyde with thickness 1 µm is deposited by means of a suitable deposition method, here by disintegration of tetraethoxysilane, $(C_2H_5O)_4Si$, at low pressure (LPCVD). figure 13, on the smooth surface 10, resulting in an overall surface roughness less than 0.15 µm. On the inorganic layer 4 a photo-lacquer layer 5 is formed with a thickness of about 1 µm, figure 14, by means of 'spin-coating'. The photo-lacquer layer 5 is then exposed to a regular pattern with the use of a mask 6. The pattern of the mask is made here of square fields with a size of 1 to 1 µm. The focusing depth of the projection system used is here at least 1 µm, it should be larger than the sum of the thickness of the photo-lacquer layer and the surface roughness of the inorganic layer 4. Subsequently the lacquer layer 5 is developed and etched leaving a reduced(5x) mask pattern of photo-laquer 5 on the inorganic layer 4, according to standard lithographic methods. In the inorganic layer 4 then the reduced mask pattern is etched by a suitable etchant, forming perforations with a square cross-section of 0.2 by 0.2 µm, herewith forming the inorganic membrane layer 4, figure 15. In this example the inorganic layer 4 of silicondioxide is etched by means of a stabilized fluoro-hydrogen (HF) solution. The etching stops as soon as the surface 10 of the support has been reached. Excellent cylindrical perforations may also be etched by means of anisotropic reactive ion etching. Next the flattening material 7 is removed from the pores in the support 1 by means of a material selective etchant that is brought into contact with the flattening material 7 through the perforations of the yet formed inorganic membrane layer 4, figure 15, 16. As a selective etchant, capable of etching very well polysilicon and hardly silicondioxide or alumina a plasma may be used containing at least oxygen in an amount of 1-10 % per mol and chlorine (Cl) in an amount of 25-30 % per mol.

From the above it may be clear that the present invention is not limited to the mentioned examples, but that for the skilled specialist many variations of the invention will be possible. For instance, for the material of the inorganic membrane layer 4 other inorganic components like sodiumoxyde, potassiumoxyde, calciumoxyde, magnesiumoxyde or silicon, gallium, arsenic, or other semiconductors and metals may be applied. The inorganic layer 4 or the support 1 may also be constituted from or coated with a biocompatibel material. Other materials than polysilicon and aluminium are also possible for the

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use of a good flattening material 7, for example a at low temperature liquifying glasslayer. The use of the flattening material 7 for obtaining a flat and smooth surface may also be circumvented, for example through a partial liquification of the inorganic layer 4. Components may also be applied in enhancing the joining strength and temperature durability between the support 1 and the inorganic membrane layer 4, for example borax and diphosphoruspentoxyde. The formed pattern in the lacquer layer 5 and subsequently in the membrane layer 4 is not limited to the use of an external mask, but may also be shaped by means of a interference pattern or with the use of a modulated laser beam. The invention is also not restricted to the use of optical lithography, other techniques with higher resolution (submicron), like Electron Beam and X-Ray Lithography are likewise appropriate.

Claims

1. Inorganic membrane for microfiltration, wherein the membrane consists of an inorganic macroporous support having a mean pore size in the range of 0.25µm-25µm, and an inorganic membrane layer being deposited and connected rigidly to a flattened surface of the macroporous support by means of a deposition method, whereby the pores of the membrane layer are perforations shaped as shallow channels square to the membrane layer, and are formed with a diameter in the range of 0.05-5µm by means of etching, said channels having a length at least smaller than ten times the diameter of the channels.

2. Inorganic membrane layer according to claim 1, whereby the support is composed of a ceramic substance.

3. Inorganic membrane layer according to claim 2, whereby the membrane layer is also composed of a ceramic substance.

4. Inorganic membrane according to claim 1,2 or 3, whereby the inorganic membrane layer comprises one of the substances from the group of silicondioxide, siliconnitride, siliconcarbide, alumina, zirconiumoxide, titaniumoxide, titaniumnitride, gold, silver, chromium, cobalt, nickel, aluminium, titanium, chromium alloys, carbon and silicon.

5. Inorganic membrane according to claim 1, 2, 3 or 4, whereby the inorganic macroporous support comprises one of the substances from the group of silicondioxide, siliconnitride, siliconcarbide, alumina, titaniumnitride, titanium, carbon and silicon.

6. Process for production of an inorganic membrane for microfiltration, the membrane consisting of a macroporous inorganic support and an inorganic membrane layer according to any of the preceding claims 1-5, whereby an inorganic layer is deposited on a flattened surface of the support by means of chemical vapour deposition or sputtering, and whereby on the inorganic layer a photo-lacquer layer is formed, this photo-lacquer layer being exposed to a regular mask pattern with the use of a suitable source, this lacquer layer being developed, thereafter in the inorganic layer the mask pattern is etched by an etchant, forming perforations shaped as channels, square to the inorganic layer, thus forming the inorganic membrane layer.

7. Process according to claim 6, whereby before the inorganic layer is deposited on the flattened surface of the support, primarily a flattening material is deposited on a surface of the support, filling all pores lying at the surface of the support, which flattening material will at least be partially removed after the forming of the inorganic membrane layer.

8. Process according to claim 6 or 7, whereby the flattening material is polysilicon, silicon or aluminium.

Patentansprüche

1. Anorganische Membran fuhr Mikrofiltration, wobei die Membran aus einem anorganischen makroporösen Träger mit einer mittleren Porengröße in der Größenordnung von 0.25µm-25µm besteht und einer anorganischen Membranschicht, welche auf einer abgeflachten Fläche des makroporösen Trägers mit Hilfe eines Auftragverfahrens aufgetragen und fest verbunden ist, wobei die Poren der Membranschicht Perforationen in der Gestalt flacher Kanäle quer zur Membranschicht sind, und mit einem Durchmesser in der Größenordnung von 0.05-5µm durch Ätzen geformt sind, besagte Kanäle eine Länge aufweisen, die mindestens kleiner als der zehnfache Durchmesser der Kanäle ist.

2. Anorganische Membranschicht nach Anspruch 1, wobei der Träger aus einem keramischen Stoff besteht.

3. Anorganische Membranschicht nach Anspruch 2, wobei die Membranschicht ebenfalls aus einem keramischen Stoffbesteht.

4. Anorganische Membran nach Anspruch 1, 2 oder 3, wobei die anorganische Membranschicht aus einem der Stoffe aus der Gruppe der Siliziumoxide, Siliziumnitride, Siliziumkarbide, Aluminiumoxide, Zirkoniumoxide, Titanoxide, Titannitride, Gold, Silber, Chrom, Kobalt, Nickel, Aluminium, Titan, Chromlegierungen, Kohlenstoffund Silizium besteht.

5. Anorganische Membran nach Anspruch 1, 2, 3, oder 4, wobei der anorganische makroporöse Träger aus einem der Stoffe aus der Gruppe der Siliziumoxide,

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Siliziumnitride, Siliziumkarbide, Alumiumoxide, Titannitride, Titan, Kohlenstoff und Silizium besteht.

6. Verfahren zur Herstellung einer anorganischen Membran fuhr Mikrofiltration, wobei die Membran aus einem makroporösen anorganischen Träger und einer anorganischen Membranschicht nach irgendeinem der vorgenannten Ansprüche 1-5 besteht, wobei eine anorganische Schicht auf einer abgeflachten Fläche des Trägers durch chemisches Aufdampfen oder Sputtern aufgetragen wird, und wobei auf der anorganischen Schicht eine Photo-Lackschicht gebildet wird, diese Photo-Lackschicht mittels einer geeigneten Quelle zu einem regelmäßigen Maskenmuster belichtet, diese Lackschicht entwickelt wird, daran anschließend in die anorganische Schicht das Maskenmuster mit Hilfe eines Ätzmittels geätzt wird, Perforationen bildet, die die Gestalt von Kanälen haben und quer zur anorganischen Schicht verlaufen und auf diese Weise die anorganische Membranschicht bilden.

7. Verfahren nach Anspruch 6, wobei, bevor die anorganische Schicht auf der abgeflachten Oberfläche des Trägers aufgetragen wird, erst ein glättendes Material auf einer Oberfläche des Trägers aufgetragen wird, das alle an der Oberfläche des Trägers liegenden Poren füllt, welches glättende Material nach der Bildung der anorganischen Membranschicht zumindest teilweise entfernt wird.

8. Verfahren nach Anspruch 6 oder 7, wobei das glättende Material Polysilizium, Silizium oder Aluminium ist.

Revendications

1. Une membrane inorganique pour la microfiltration, où la membrane consiste en un support inorganique macroporeux avec des pores d'une taille moyenne de l'ordre de 0,25 µm - 25µm et en une couche de membrane inorganique appliquée et connectée rigidement sur une surface aplatie du support macroporeux au moyen d'une méthode d'application, les pores de la couche de membrane étant des perforations formées comme des canaux superficiels à angle droit avec la couche de membrane, et étant formés avec un diamètre de l'ordre de 0,05 - 5 µm par gravure à l'eau forte, ces canaux ayant une longueur au moins inférieure à dix fois le diamètre des canaux.

2. Une couche de membrane inorganique d'après la revendication 1, où le support est composé d'une substance céramique.

3. Une couche de membrane inorganique d'après la revendication 2, où la couche de membrane est également composée d'une substance céramique.

4. Une couche de membrane inorganique d'après la revendication 1, 2 ou 3, où la couche de membrane inorganique comprend l'une des substances du groupe bioxyde de silicium, nitrite de silicium, carbure de silicium, alumine, oxyde de zirconium, oxyde de titane, nitrite de titane, or, argent, chrome, cobalt, nickel, aluminium, titane, alliages de chrome, carbone et silicium.

5. Une couche de membrane inorganique d'après la revendication 1, 2, 3, ou 4, où le support inorganique macroporeux comprend l'une des substances du groupe bioxyde de silicium, nitrite de silicium, carbure de silicium, alumine, nitrite de titane, titane, carbone et silicium.

6. Un procédé pour la production d'une membrane inorganique pour la microfiltration, la membrane consistant en un support macroporeux inorganique et en une couche de membrane inorganique d'après toutes les revendications précédentes 1 à 5, une membrane inorganique étant appliquée sur une surface aplatie du support au moyen de l'application d'une vapeur chimique ou par projection, une couche de laque photo se formant sur la membrane inorganique, cette couche de laque photo étant exposée à un motif masquant régulier en utilisant une source adaptée, cette couche de laque étant développée, le motif masquant étant gravé ensuite à l'eau forte par une pointe dans la couche inorganique, formant des perforations formées comme des canaux, à angle droit avec la couche inorganique, formant ainsi la couche de membrane inorganique.

7. Un procédé d'après la revendication 6, où, avant que la couche inorganique soit appliquée sur la surface aplatie du support, un matériau aplatissant est d'abord appliqué sur une surface du support, remplissant tous les pores se trouvant à la surface du support, ce matériau aplatissant étant au moins partiellement enlevé après la formation de la couche de membrane inorganique.

8. Un procédé d'après la revendication 6 ou 7, où le matériau aplatissant est du polysilicium, du silicium ou de l'aluminium.

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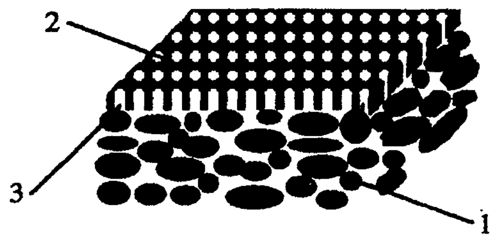

Figure 1

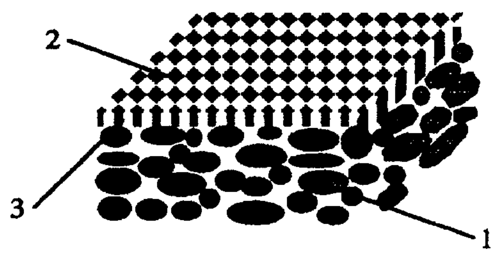

Figure 2

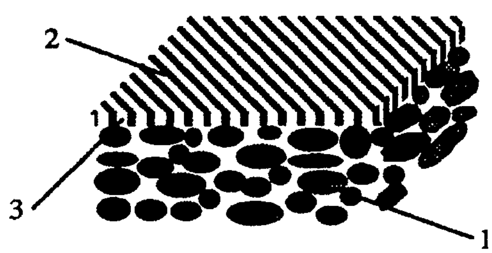

Figure 3

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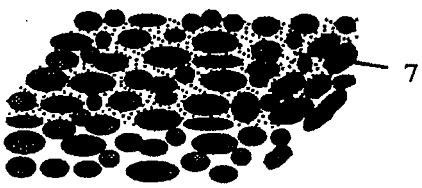


Figure 4

Figure 5

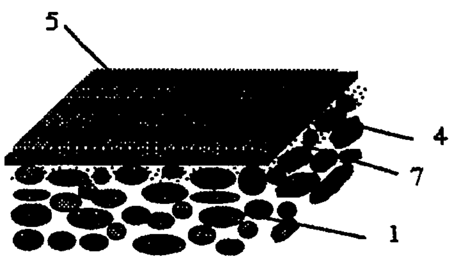


Figure 6

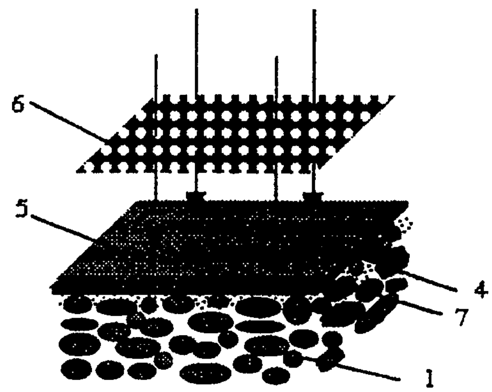


Figure 7

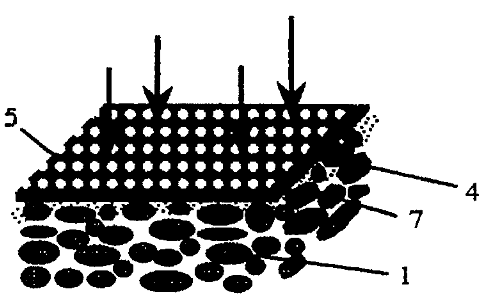


Figure 8

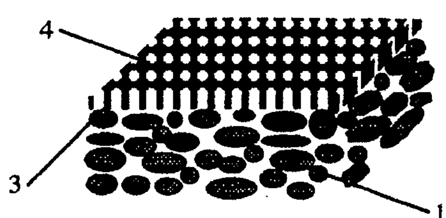


Figure 9

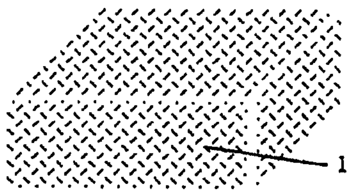


Figure 10 a

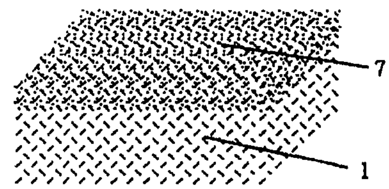


Figure10 b

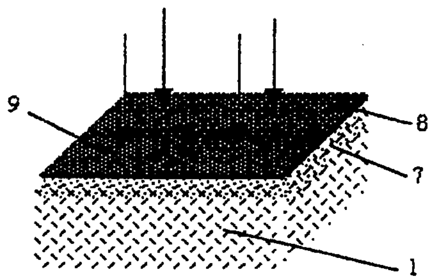
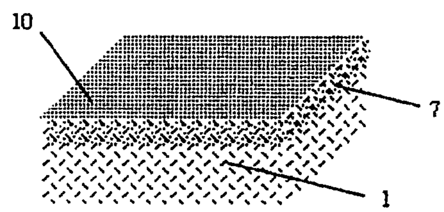


Figure 11



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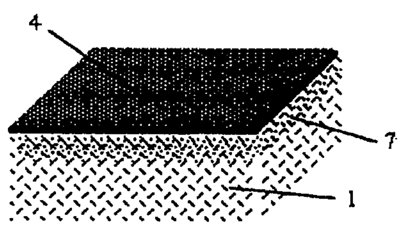

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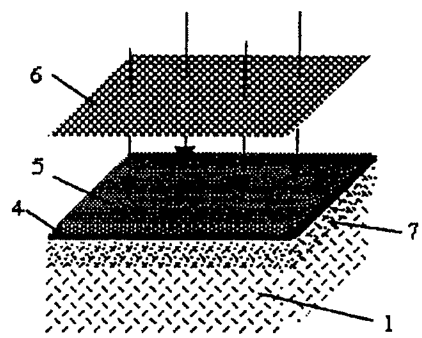

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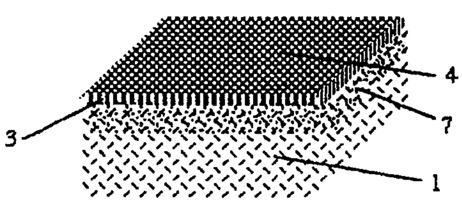

Figure 15

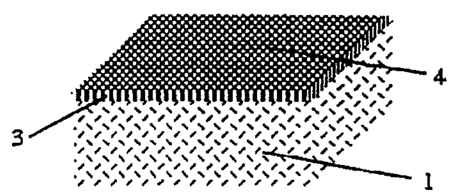

Figure 16

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